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Measurement of weak non-linear response of Kevlar® fibre damaged by UV exposure

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ABSTRACT

This paper deals with a high-sensitivity method for the assessment of damage in high-strength fibres exposed to UV radiation. A recently developed experimental testing machine, based on an optical measurement system and electro-magnetic driving force, was used to characterize fibre materials. Stiffness, damping, and non-linearity were measured on several Kevlar® fibre samples previously exposed to UV light for different lengths of time. The results show that UV radiation increases the material non-linearity by amounts which can be clearly observed even at low vibration amplitudes. On the contrary, uncertainties affecting the determination of stiffness and damping with the adopted approach don't seem to allow a similarly unambiguous UV damage assessment.

Result confirm our initial hypothesis that non-linearity may be a valuable index of damage, at least in case of UV exposure, for applications in the Structural Health Monitoring (SHM) field.

Keywords: *non-linear response; vibration based monitoring; damping; dynamic response; UV degradation; high-strength fibres.*

1 INTRODUCTION

High-strength materials have applications in a great variety of engineering sectors: aerospace, civil, automotive, technical clothing, defence or sport equipment, and are exposed as a consequence to a variety of external (environmental) factors, which may degrade their properties. Effects may be cumulative over the life of the structural element. Materials and design should then be planned bearing in mind these conditions, in order to maximize benefits for structure reinforcement purposes. In the specific area of structural engineering and retrofitting [1], problems arising from the structural performance of polymeric materials after long-term exposure to the agents make the end-users cautious as to when and where to apply them [2]. Systems using high-strength materials can be suitable for every need due to the possibility of having different FRP configurations, material properties, and installations. However, when high-strength polymeric fibres are used as

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retrofitting materials they may be externally glued to structures and thus exposed to ultraviolet (UV) radiation [3,4]. Such radiation is dangerous for any polymer, because it accelerates corrosion processes induced by native oxygen, resulting in weight loss and deterioration [5]. In particular, UV radiation with wavelength from 200 nm to 400 nm can degrade the resin matrix in which fibres are embedded, and can even degrade the fibres themselves after excessive exposure.

Aramid materials are highly UV-sensitive and excessive UV exposure results in reduction of the ultimate tensile strength of fibres because of depolymerisation and chain breaking. The main bonds in aramid fibres are C–O and N–H, whose corresponding bond energy resonance wavelengths are 340 and 306 nm, respectively. Consequently, the required wavelength to break the N–H bond is 306 nm. After UV exposure, micro cracks on the fibre surface appear.

Effects on the stress-strain curve have been also observed in the work presented by Huang Gu [5]: the typical stiffening stress-strain curve of aramid fibres is replaced by a bilinear softening response with a yielding point in seriously degraded samples [6]. Investigations on the ultimate resistance were carried out also for high UV exposures especially in the context of space applications where a loss of about 20% of tensile strength and 30% ultimate strain was experienced for Kevlar® and even higher values were observed for other high-strength fibres such as Vectran®, Spectra® and Zylon® [7]. Other studies, such as [8], have reported results on UV degradation of thick ropes. The work presented by Said et al. [4] studied high-strength fibres in single continuous yarn form, and quantified their strength loss with UV exposure. This work further claimed that Kevlar® is self-screening. In fact, its stability with light exposure depends on the thickness of the exposed item. Very thin Kevlar® 49 fabric, if directly exposed to very high intensity sunlight for an extended period, will lose about half the tensile strength within a few days. In thicker elements, such as a half-inch diameter rope, the outer layer protects most of the rope and strength loss is minimized.

The present paper stems from a collaboration between the Structural Engineering and the Metrology research groups of Politecnico di Torino, which yielded a prototype testing machine designed ad hoc to quantify the mechanical damping of fibre materials and the influence of weaving on the fibre behaviour. The design of the machine is extensively described in a recently published paper [9] where a relatively low-cost method was proposed to measure material properties such as damping, non-linearity and tensile modulus of several types of fibres. The idea of this work is that certainly a decrease in ultimate tensile strength can be expected to induce increased non-linearity at low tension levels, and a specific inquiry may then be desirable to assess if UV damage can be efficiently monitored by detection of increased non-linearity, via strength reduction [3-5]. To this aim, material properties of Kevlar samples were evaluated after various UV exposure times with the resonant small oscillations approach, by employing the Politecnico test machine. The experimentally identified correlation between strength degradation and non-linearity is a novel result which opens the way to promising applications of the methodology in non-destructive diagnosis of UV degradation in aramid fibres.

The paper is structured in a methodology section and a results and discussion section. In the first, a simple but reliable UV damage procedure is defined for aramid fibres, followed by a short description of the experimental machine. Vibration test procedure and estimation of the non-linear backbone curve are also discussed. In the second, results obtained for the different exposure levels are presented, with the aim to identify a trend in material properties as a function of total exposure.

2 METHODOLOGY

2.1 UV damage procedure

The procedure chosen to artificially damage aramid fibre samples is prolonged exposure to UV radiation. Continuous and prolonged exposure is more harmful than an intermittent one, because the dangerousness of the attack depends on the extent and the degree of exposure. The polymers that possess UV-absorption groups, such as aromatic rings (aramid and para-aramid), can be sensitive to the effect of ultraviolet radiation, and should be protected from the deleterious effects of sunlight. The damage caused by UV effects on this type of materials, which is visible after a long-time exposure, can be evaluated by means of accelerated exposure testing. In order to carry out this kind of test, a UV lamp for industrial applications (OSRAM Ultra Vitalux®) was chosen [10]. The main technical characteristics of the UV lamp are listed in Table 1.

Table 1. Technical details about Osram Vitalux®.

Electrical data	
Nominal power [W]	300
Nominal voltage [V]	230
Photometric data	
UVA emission [W] (after 1 hour of operation)	13.6
UVB emission [W] (after 1 hour of operation)	3
Geometric size	
Diameter [mm]	127
Length [mm]	185

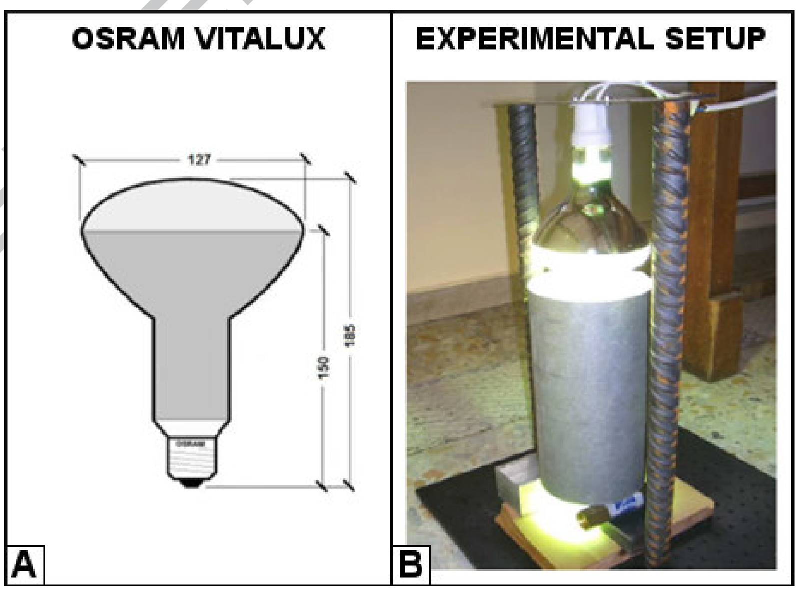

Figure 1. Technical details of Osram Vitalux lightbulb for scale comparison (A) and the experimental setup used to guide the light flux onto fibre samples (B).

The experiment was conducted in the Metrology Laboratory of the Department of Electronics of Politecnico di Torino. The UV lamp was mounted on a support as shown in Figure 1B, with a steel tube (Ø13 cm) used to direct most of the light toward the fibre sample. An open gap below the cylinder ensured a natural air circulation during the test to avoid excess temperature increase.

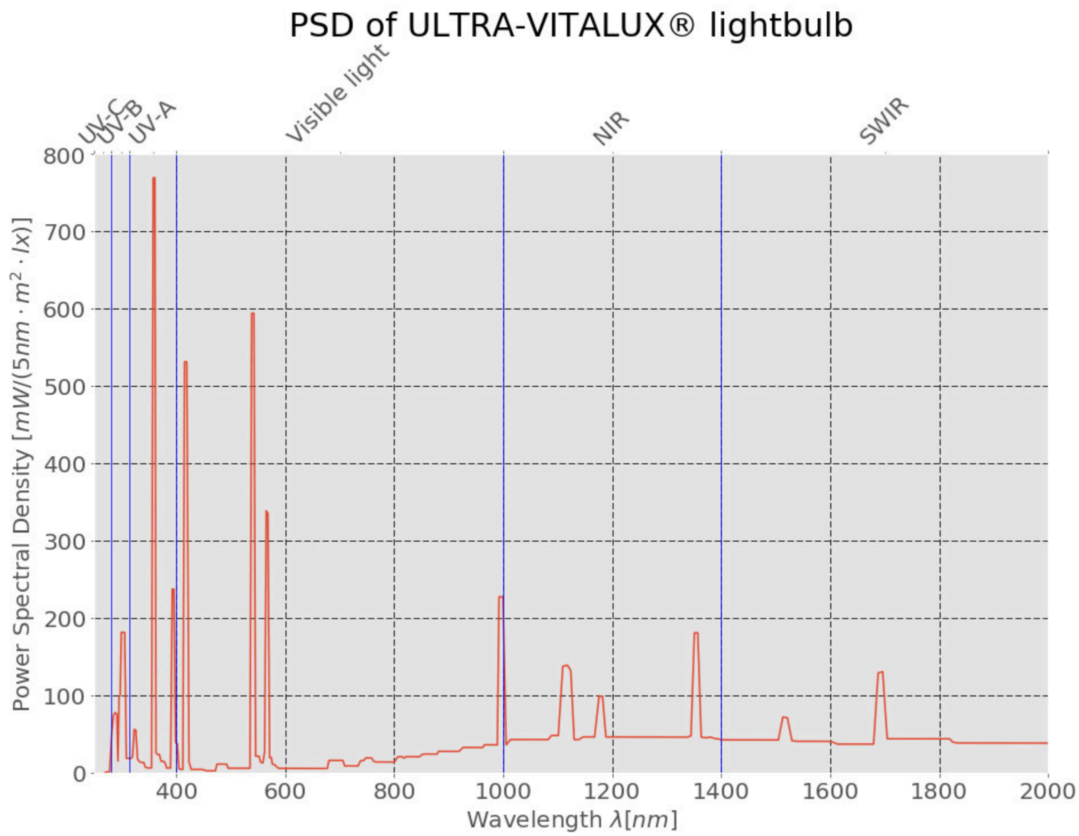

Figure 2. Power Spectral Density (PSD) of Ultra-Vitalux® lightbulb subdivided for the different components of the electromagnetic spectrum (NIR: near infrared, SWIR: short-wave infrared).

The relevant feature of the UV lamp emission spectrum, shown in Figure 2, is the bright line around 300 nm (in the UVA region) because it happens to fall exactly on resonance with the critical wavelength of the unstable N-H bond of aramid fibres. Since most of the UVA emission is in that bright line, its power content can be taken to be roughly equal to the UVA total flux specification of 13.6 W given in Table 1, which is then used here to figure out the total radiation dose per hour of exposure. It is here assumed that the guiding efficiency for UV of the steel tube shown in Figure 1B be rather good, so that most of the flux would flood the target down below. Once a week, the sample was turned upside down to produce as uniform as possible a damage. A flux of roughly 1 kW/m^2 is then assumed on the fibre samples, which means about 6 mW/m on a single 12 μm diameter fibre, considering an average screen effect of 50% [4]. The total UVA dose on 1 m of a single fibre would then be about 20 J per hour of exposure. Clearly, these evaluations are not very precise, but they can certainly be taken as a reasonable indication of what can be expected in real-life exposure, given the fact that average sunlight UVA flux values are also quite uncertain in real life. Since the UVA fraction of sunlight reaching the Earth is approximately 5% and a typical clear sky total irradiance is about half a kW/m^2 at its peak, it can be safely stated that the used UVA flux was roughly 40 times the real-life average cumulative exposure that fibres may have to face in operation.

Three different samples were continuously exposed to UVA radiation from the lamp for 792, 1272, and 2040 hours respectively. Such times of accelerated test roughly correspond to 8, 13, and 20 years of exposure to sunlight in temperate latitudes, considering 8 hours per day of sunlight.

2.2 Testing-machine and characterisation methodology

A technical review of the testing-machine used in this work was published recently [9]. An extensive review on system identification methods in structural dynamics can be found in the literature [11]. The method chosen was selected with the aim of achieving high-sensitivity. A dynamic resonant approach is adopted to measure damping, dynamic elastic modulus, and non-linearity of fibre materials in a spring-mass system, excited by a sine-wave forcing term, in which the material under test is the spring. The prototype testing machine is shown in Figure 3, and consists of a portal with an upper beam from the centre of which the fibre specimen is hanging, holding the mass attached at the bottom. The mass is constrained to the single vertical degree of freedom by a harness of fibre tie-rods. The forcing term is applied vertically to the suspended mass by a simple voice-coil made of a 50 mm wide NdFeB magnet driving 1 Tesla through a 6 mm gap, and a single horizontal wire rigidly connected to the mass. The induced vertical vibrations are detected by a contactless optical sensor, made with a ball lens and a split detector. The aim is to minimize energy dissipation and allow accurate observation of the intrinsic damping of the fibre material.

The apparatus can be used in the free ring-down mode, but most of the measurements were carried out in the forced oscillation mode, by operating it at various frequencies around resonance and plotting response versus frequency. The resonance frequency itself is accurately identified from the phase difference between forcing term and oscillation, which is 90° right there, and can be used to evaluate the tensile module. The damping is obtained from the measured resonance quality factor (Q) using the canonical half-power bandwidth method, and non-linearity is characterized by a dimensionless index which is evaluated through the identification of the “backbone curve” in a family of resonance curves obtained at different levels of excitation. This method turned out to be so sensitive as to allow rather precise evaluation of the non-linearity index even at excitation levels as low as a few percent of the ultimate strength of the sample.

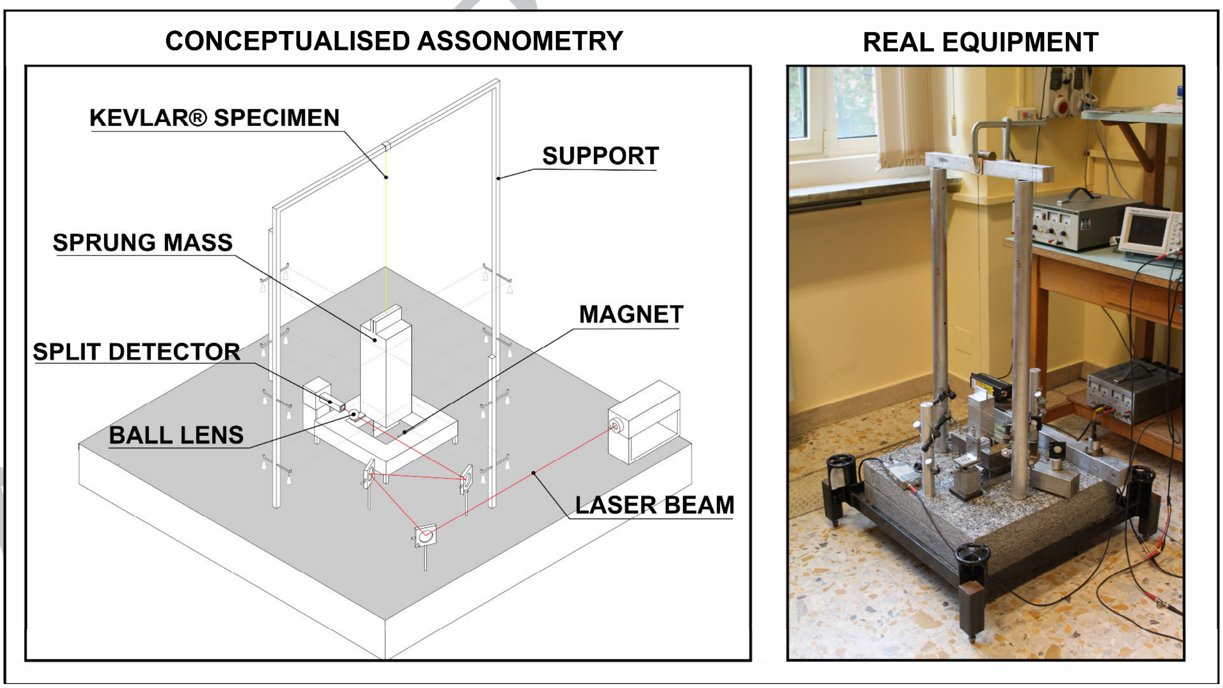

Figure 3. Schematic of the optical test system, the resonant system and the support frame.

The suspended 1.375 kg aluminium block has the role of the mass in the spring-mass system. Its size is chosen as to keep the sample in tension, typically at 10% of its tensile strength, so that resonant measurements may be carried out without fully unloading the sample. The sinusoidal excitation was applied to the sample through the voice coil by a function generator (AGILENT 3320A), with 1 μHz resolution in the range of interest. Amplitudes of the induced vertical oscillation sine wave were measured with an AC digital Voltmeter. Given the 50 Ohm output resistance of the generator, the short circuit output current of 200 mA peak-to-peak delivered at the maximum peak-to-peak amplitude of 10 V is within the specified linear range. Nevertheless, a check on distortion was run for safety before deciding to load directly the generator with the low resistance voice coil.

The frequency range investigated was of ±0.2 Hz around the system resonance. Because of the long time-constants determined by high Q values at low frequencies, the sweep-sine approach was avoided in favour of measuring the steady-state response with discrete steps of 10 mHz. About 40 points were taken for each response curve. The test was repeated with different levels of the driving force, regulated by adjusting the current driven into the voice coil by the function generator, in steps of 20 mA from 40mA to 200 mA peak-to-peak, corresponding to a maximum rms force range of about 3.5mN and 0.35mN steps. The rms displacement response was determined by using the detector sensitivity measured for each curve in DC during photodiode calibration.

2.3 Non-linear response estimation using Krylov-Bogoliubov method

Following the findings of the previously published set of experiments [9], a cubic non-linear behaviour was observed in the fibre specimen. The dynamics of a system characterized by cubic non-linearity excited by a harmonic force was modelled by the Duffing oscillator equation:

$$m\ddot{x} + c\dot{x} + kx + k_3 x^3 = p(t) \tag{1}$$

where $p(t)$ is the external sinusoidal forcing term, m the mass of the system, c the linear viscous damping term, k the linear stiffness term and k_3 the cubic stiffness coefficient, respectively. In order to identify the non-linear material behaviour, the Krylov-Bogoliubov method was used to determine the variation of the first non-linear resonance frequency of a Duffing oscillator of Equation (1), characterized by a non-linear force-displacement relationship [12,13]. The resonant frequency distortion can be quantified by the non-linear backbone curve connecting peak amplitudes a_p of the various resonance curves at different excitation levels. The frequency $f(a_p)$ at which the peak occurs can be approximated using perturbation analysis [14] as

$$f(a_p) = f_0\sqrt{1 + \frac{3}{4}\frac{k_3}{k}a_p^2} \tag{2}$$

In order to identify a material-specific device-independent parameter, the backbone curve will be represented here in terms of the deformation strain ε, with a dimensionless coefficient $X = (3/4) L^2 k_3/k$ dimensionless and suitable to characterize material non-linearity. Its rms value, indicated just as ε, will be used for consistency. Equation (2) is then rewritten as

$$f(a_p) = f_0(1 + X\varepsilon^2) \tag{3}$$

f_0 being the frequency of the underlying linear resonance. The common engineering unit με will be adopted, usually indicated as "micro-strain" (1 με =1μm/m). Equation (3) was used to identify X from the backbone curve.

This approach confirmed the high sensitivity of this new prototype-testing machine to the dynamic characterization of high-strength materials, even in presence of low levels of non-linearity [9]. Indeed, the non-linear coefficient was measured even for very small intrinsic non-linearity in the material samples (like in the pristine sample below).

3 RESULTS AND DISCUSSION

Tests were carried out on different Kevlar®-29 yarn samples extracted from the same bundle. Yarns consist of roughly 1500 fibres, 12 µm in diameter, judging from the specified 2500 dTex number provided by the manufacturer, which yields a total cross-sectional area of 0.174 mm² for the bundle. This value is used to convert voice coil force into applied stress, for which a maximum rms value of about 20 kPa is obtained. Four samples were cut to equal length, for a free length in operation in the test machine of 665 mm. Uncertainty on the latter was estimated to be about ±1 mm. One of the samples was not exposed to UV and served as a reference. The other three were exposed to radiation as detailed in section 2. The yarn is pale yellow when new, but takes on an increasingly "burnt" colour after exposure to UV radiation.

The resonance frequency of about 20 Hz of all samples returns a tensile modulus of 70 GPa by using the mentioned values for length and cross section, quite in line with typical data which can be found in the literature. Sample-to-sample variations are below 5% and must be considered meaningless due to lack of information on actual fibre count and diameter.

The damping coefficient ζ (estimated with the half-power bandwidth method) appeared to vary for all samples with the excitation level, increasing from below 0.005 at the 4 kPa rms (quality factor Q above 100), to below 0.006 at the 20 kPa rms (Q below 80). However, values obtained with this approach are quite strongly affected, for low excitation, by the judgement on where the zero is positioned in the curve, and for high excitation by detector saturation. Uncertainty must therefore be taken conservatively to be at least of the order of a few percent, and higher at low excitation. A correction can be applied to account for detector non-linearity, which seems to suggest smaller dependence of damping on excitation level than is shown in Figure 4. This is not reported here as quality factor at the end didn't turn out to change significantly with UV exposure and was finally discarded as a useful indicator for UV damage in Aramid fibres.

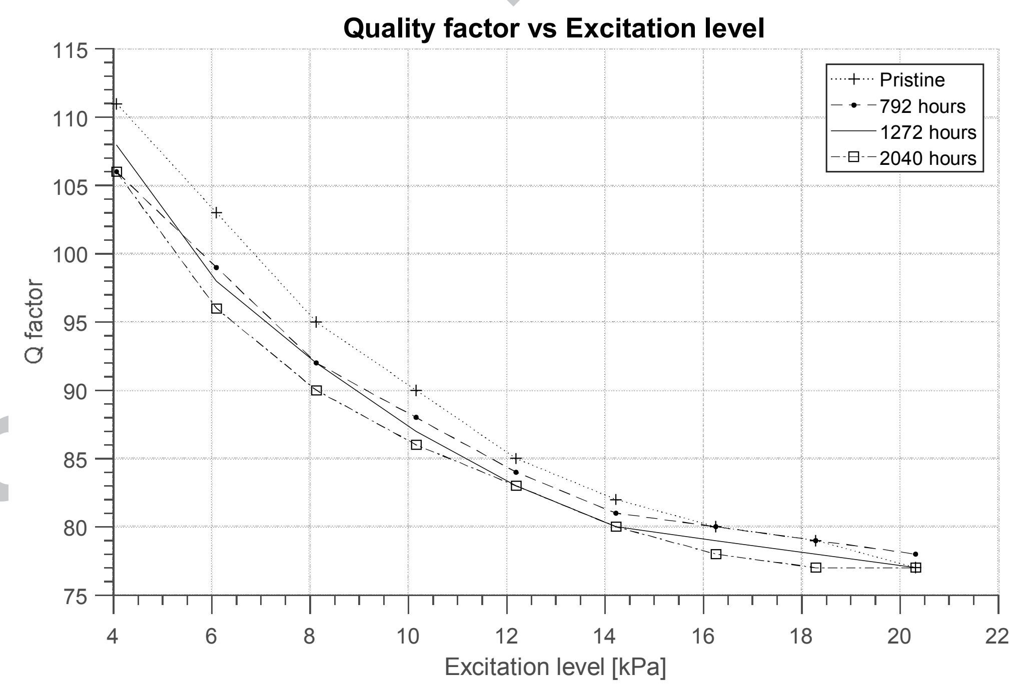

Figure 4. Measured variations of Q-factor versus drive level for different exposure times to UV.

In Figure 4, variations with excitation level of measured quality factor Q are shown for all four samples. It can be noticed that the curves show similar magnitude and behaviour. Because of the uncertainty in the Q factor determination, the slight decrease in the Q values that might be inferred by these curves does not appear to be significant.

It may be useful to point out here that the peak tension developed on resonance in the sample for the maximum excitation stress of 20 kPa rms, as obtained by multiplying it by the Q factor, can be inferred to be about 2.2 MPa. This is less than 3% of the preloading given by the weight of the oscillating mass at rest (which guarantees the sample is never fully unloaded), and less than 0.1% of the ultimate strength of the material (which proves the ability of the adopted approach to monitor damage without overstressing the sample).

Response curves measured for all samples are shown in Figure 5 to 8. The bottom curve corresponds to the minimum driving stress of 4 kPa rms (40mA$_{pp}$ from the generator), while the top one corresponds to the maximum applied driving stress of 20 kPa rms (200mA$_{pp}$ from the generator). The step of driving stress from one curve to the next is 2 kPa rms (20 mA$_{pp}$). The rms strains were calculated by using the applicable sensitivity value as measured for the specific experiment in the preliminary DC calibration of the optical position sensor. Values ranged from 50 to 100 mV/µm. It appears obvious by inspection when looking at these families of curves that non-linearity is dramatically increased by UV exposure.

The coefficient X of Equation (3) was estimated for each sample by fitting the theoretical parabolic shape of the backbone curve onto the family of resonance curves. The dots mark the peaks of response curves and the dashed red line is the best estimated backbone curve. The coefficient X was obtained by peak frequencies and peak strains of various pairs of curves in Equation (4), and combining results in an uncertainty weighted average.

$$\frac{f_1-f_2}{f_2} = X(\varepsilon_1^2 - \varepsilon_2^2) \tag{4}$$

Here indexes 1 and 2 identify parameters of the two curves of the pair used in the calculation. Absolute uncertainty was evaluated for X to be less than $1\cdot10^{-6}$ for all families of curves by computing the standards deviation of such values and dividing it by the square root of the used number of pairs. This means for the most damaged sample a relative uncertainty of roughly 5%, aligned to what evaluated for the other quantities. However, in this case, it turns out to be more than adequate to support clear evidence of the effects of UV exposure because X was increased in the process by almost a factor of four.

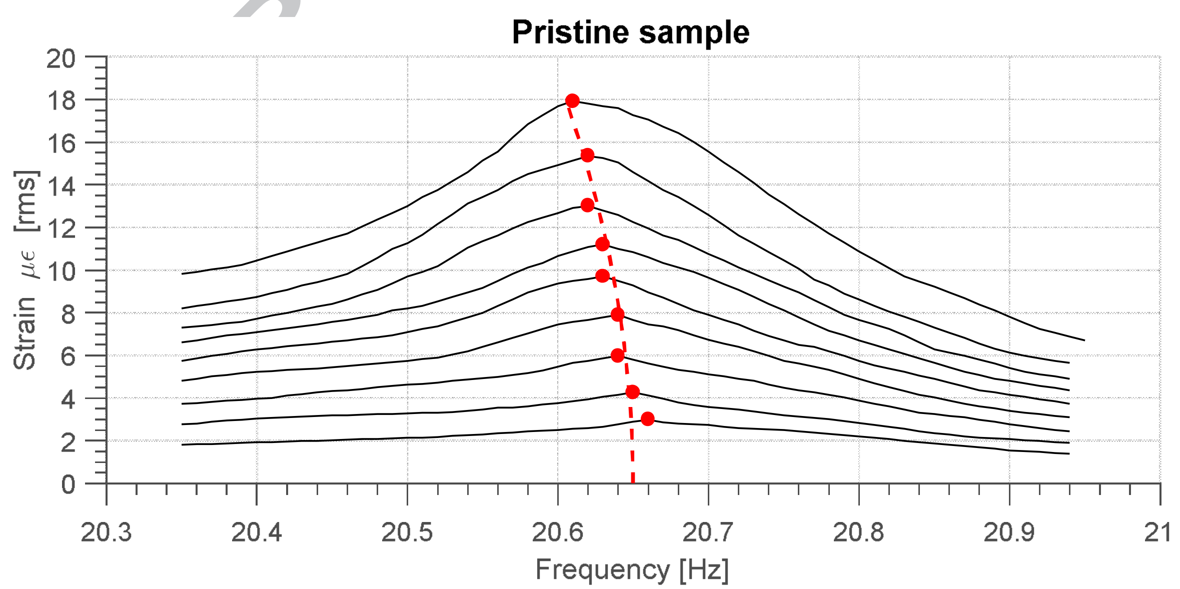

Figure 5. Pristine sample: resonance shape for various levels of rms driving stress.

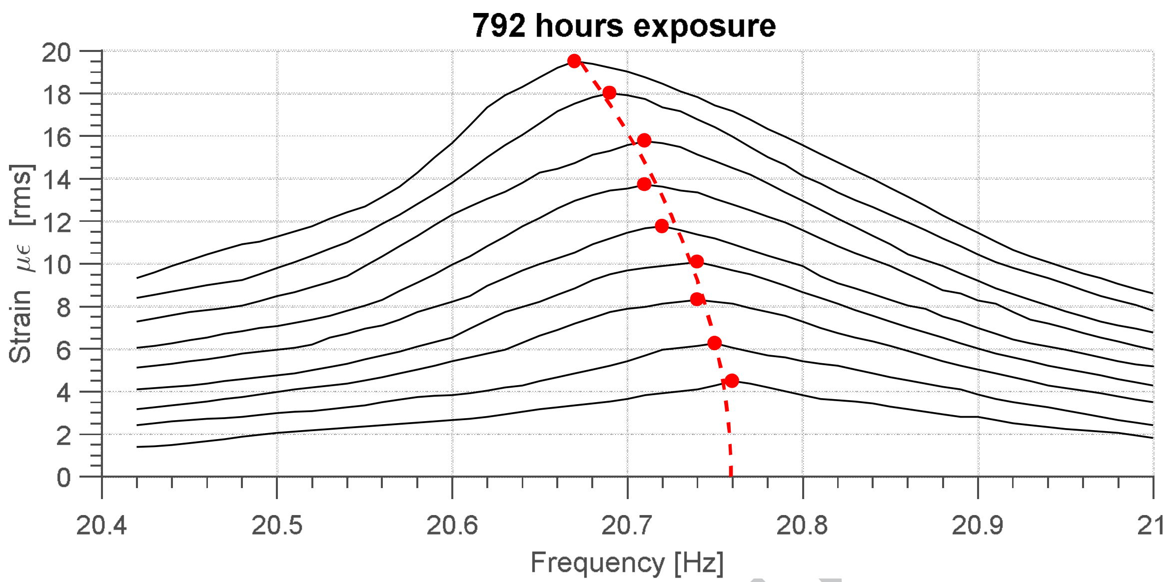

Figure 6. Sample exposed 792 hours: resonance shape for various levels of rms driving stress.

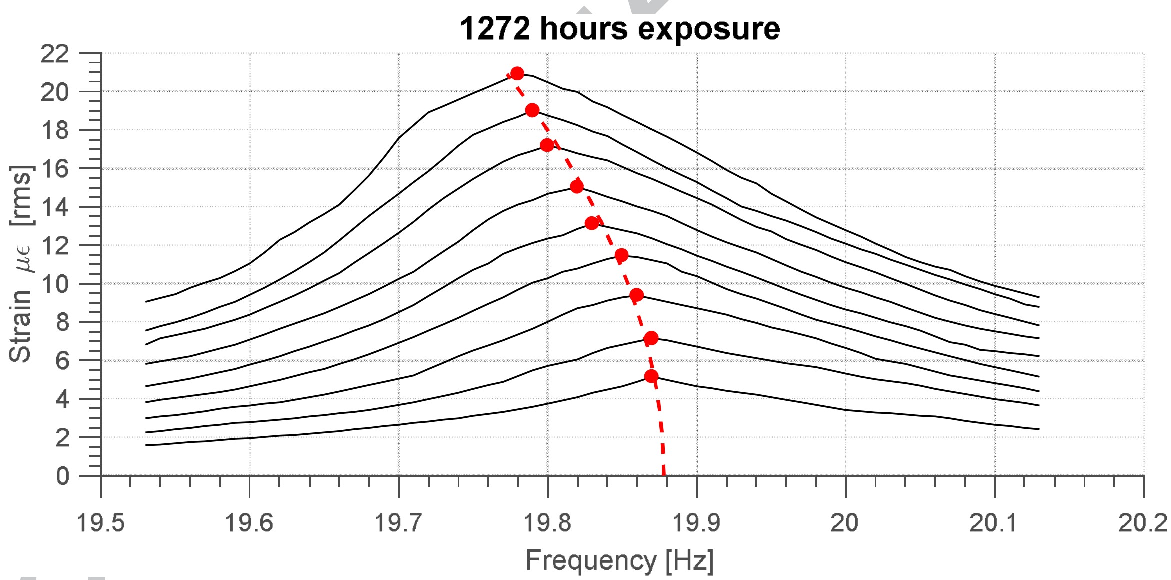

Figure 7. Sample exposed 1272 hours: resonance shape for various levels of rms driving stress.

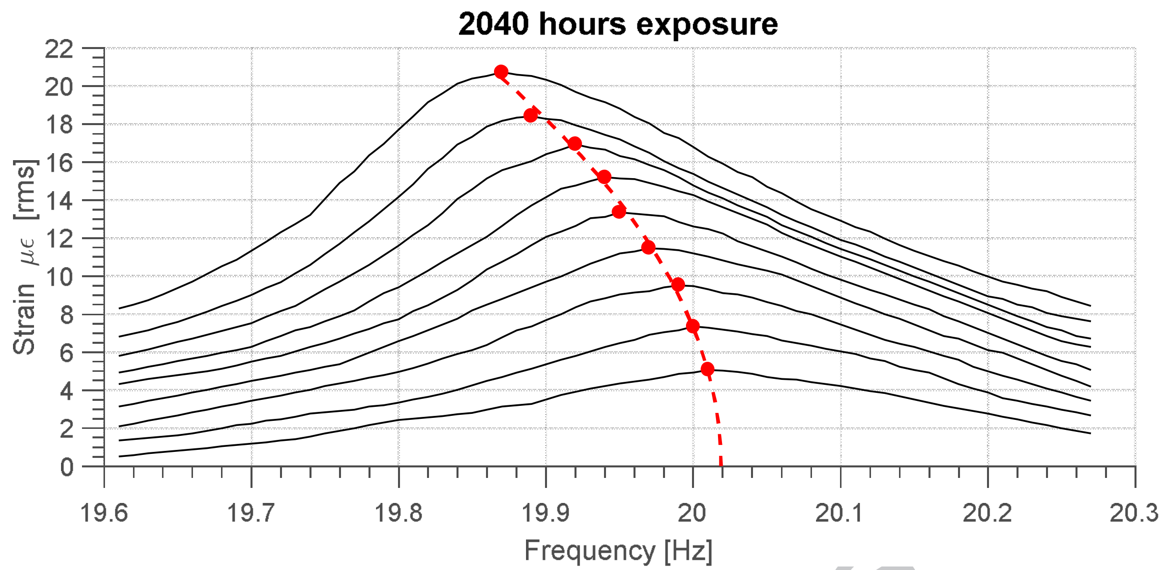

Figure 8. Sample exposed 2040 hours: resonance shape for various levels of rms driving stress.

The best values obtained for the coefficient X are $-5.6\cdot10^{-6}$, $-1.08\cdot10^{-5}$, $-1.41\cdot10^{-5}$, and $-1.81\cdot10^{-5}$ per square microstrain respectively for 0, 792, 1272, and 2040 hours of exposure to UV radiation. The progressive increase in non-linearity is represented in Figure 9, where a linear increase of the coefficient X versus exposure time can be clearly observed. The slope of the non-linearity coefficient change versus exposition to UVA radiation time in hours can be evaluated from this plot to be about $-6.1\cdot10^{-9}$/h at the used irradiation level of 1 kW/m^2. This is equivalent to about $-0.3\cdot10^{-9}$/J of incident UV energy on 1 m of a single 12 µm fibre, according to the dose evaluation given in Section 2. Whether the damage per Joule depends on radiation level remains to be determined. What can be safely stated here is that unambiguous detection of UV damage can be assessed with this method at the present level of uncertainty after about 150 h of continuous exposure to 1 kW/m^2 UVA radiation, because that is the time that it takes in these conditions for X to increase more than the uncertainty. This is equivalent to about 2 years of normal environmental UV exposure at mid latitudes if damage is independent of intensity. The question whether this is true deserves more experimentation.

How changes in the non-linear coefficient are physically related with fibre deterioration, and whether reduced fibre strength can be reliably evaluated by measurements of non-linearity with the low excitation method illustrate here, are questions that most certainly deserve further attention, but the possibility of developing this approach into a field applicable non-destructive method appears unquestionably attractive.

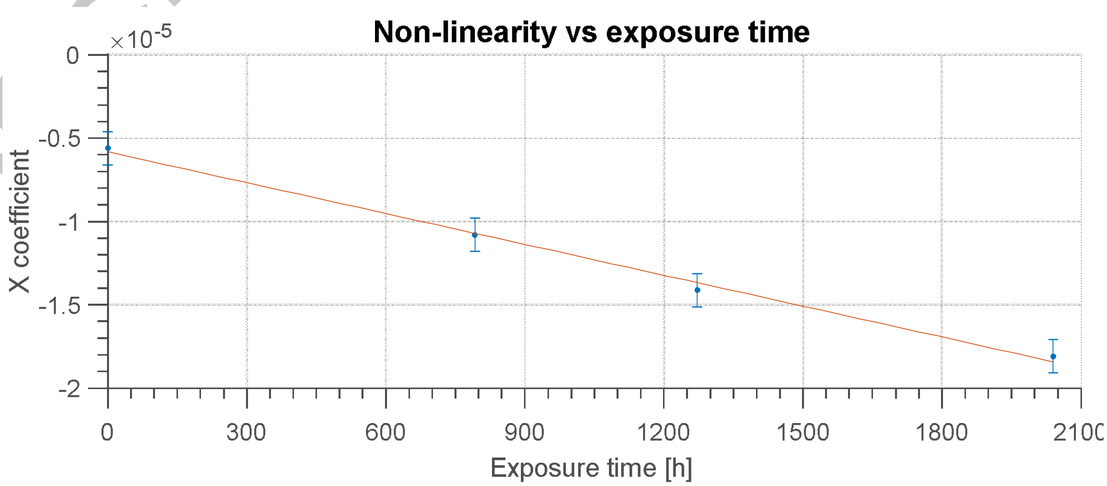

Figure 9. Variations of measured non-linear coefficient X with respect to UV exposure time.

4 CONCLUSIONS

This paper explored the possibility of assessing damage caused on high-strength fibres by long term exposure to UV radiation, with a low excitation resonant approach aimed at the measurement of material's mechanical characteristics. In particular, samples of Kevlar-29® fibres were exposed to different doses of UVA radiation, produced by a calibrated lamp, in an accelerated aging experiment. The evolution of their mechanical parameters was monitored by means of a recently realized testing machine, conceived for high-sensitivity dynamic measurements. Viscous damping, non-linearity coefficient, and potentially Tensile Modulus (through resonance frequency) can be estimated and compared between the different damage levels.

The coefficient X of Equation (3) was used to characterize non-linearity, and was evaluated with the method of fitting a parabolic backbone curve, connecting the maxima, to the family of resonance profiles taken at different excitation levels.

The experimental results showed that X is sensitive to cumulated UVA radiation, increasing linearly with the latter. Its measured variations after quite normal amount of irradiation, in fact, greatly exceed the uncertainty with which it is evaluated with the proposed method. This is undoubtedly due to a weak non-linear response of the material as all other source of non-linearity have been duly considered, modelled and quantified as negligible [9,15]. Using this parameter to assess cumulated damage occurred in Kevlar fibres from exposure to environmental UVA radiation should therefore be considered seriously.

The other characteristics that were considered do not show the same clear and easily identified sensitivity, and should therefore be discarded as damage detectors. Specifically, the tensile modulus does not seem to suffer noticeably from UV irradiation, although the increasing non-linearity suggests a possible decrease in ultimate strength. How exactly the two may be physically related to radiation appears to deserve further study at the light of results presented here.

Therefore, this result is confirming our initial hypothesis that this parameter may be used as a valuable and trustworthy index of damage in case of material evaluation, for applications in Structural Health Monitoring (SHM) field. With the uncertainty level demonstrated in this work, a couple of years of deployment in normal outdoors would already show a measurable increase in non-linearity, which would imply a decrease in ultimate strength. Clearly, more sensitive methods might be devised, allowing even earlier warning of UV damage.

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